

Europäisches Patentamt European Patent Office Office européen des brevets



(ii) Publication number:

0 405 967 A2

**(3)** 

## **EUROPEAN PATENT APPLICATION**

- Application number: 90307071.2
- 2 Date of filing: 28.06.90

(a) Int. Cl.5: **C11D** 1/825, C11D 1/835, C11D 1/83, C11D 1/86

- 9 Priority: 30.06.89 US 373912
- Date of publication of application: 02.01.91 Bulletin 91/01
- Designated Contracting States:
   AT BE CH DE ES FR GB IT LI LU NL SE
- Applicant Amway Corporation 7575 East Fulton Road Ada Michigan 49355(US)
- ② Inventor: Flower, David Michael 7232 Mount Ash, S.E. Grand Rapids Michigan 49506(US)
- Representative: Robinson, Anthony John Metcalf et al Kilburn & Strode 30 John Streeteet London, WC1N 2DD(GB)
- Built liquid detergent compositions.
- © Liquid laundry detergents comprising at least one ethoxylated nonlook surfactant, at least one alkyl polyglycoside and at least one detergent builder display excellent shelf stability, notwithstanding the fact that such compositions contain from 20 to 50 percent by weight of builder and from 5 to 22 weight percent by ethoxylated nonlooks surfactant.

EP 0 405 967 A2

Xerox Copy Centre

## BUILT LIQUID DETERGENT COMPOSITIONS

This invention relates to built liquid detergent compositions for laundry and general purpose applications which exhibit stability against phase separation.

Built liquid detergent compositions are popular with consumers because they are easy to store, dispense and measure and do not cake as some granular detergent products are apt to do when stored for lengthy periods of time or become damp. A major disadvantage of built liquid detergent compositions has been their cost in comparison to the granular products. The cost of built liquid detergents is exacerbated because of the difficulties encountered in preparing homogeneous built liquid detergent compositions.

The current commercially available built liquid detergent compositions typically contain one or more synthetic detergents and an inorganic builder, such as potassium polyphosphate, in an aqueous medium. One major problem encountered in formulating built liquid detergent compositions is the difficulty of including in a concentrated solution sufficient detergent and builder to provide the performance expected of a product intended for washing soiled clothing and other household chores. Attempts to combine the detergents and builders at effective levels in a homogeneous liquid system have resulted in the development of complicated formulae with large numbers of ingredients, including linactive cleaning ingredients such as hydrotropes, polymeric stabilizers and thickeners in these formulations. These inactive cleaning ingredients contribute little, if any, to the performance of the liquid detergent and, in fact, are sometimes deleterious to cleaning performance. They do increase the cost of the formulation and their use is justified only if it results in higher concentrations of detergent and builder in the solution.

In accordance with the present invention, there is provided a liquid built laundry detergent composition in which a high percentage of a nonionic surfactant can be incorporated with a high percentage of detergent builder without phase separation. Further, this is unexpectedly accomplished with a material which is an active cleaning agent. According to the present invention, a liquid detergent composition comprises from 5 to 22 percent by weight of at least one ethoxylated nonionic surfactant, from 3 to 11 percent by weight of at least one alkyl polyglycoside and from 20 to 50 weight percent of at least one detergent builder, and soft water.

The principal ingredients of the built liquid laundry detergents of the invention include at least one builder, at least one ethoxylated nonionic surfactant and at least one polyalkyl glycoside. These principal ingredients are included in the liquid detergent compositions in the following ranges:

Ingredient	Range	Preferred Range	Best Mode
Ethoxylated nonionic	5-22%	5-12%	7%
Alkyl polyglycoside	3-11%	4-9%	7%
Builder	20-50%	30-45%	35-45%

The Alkyl Polyglycosides

The alkyl polyglycosides which are employed as a cosurfactant in this invention are preferably those having a hydrophobic group containing from about 6 to 30 carbon atoms, preferably from about 10 to 16 carbon atoms and a polysaccharide, for example, a polyglycoside, hydrophylic group containing from about 1-1/2 to about 10, preferably from about 1-1/2 to about 3, and most preferably from about 1.8 to about 2.7 saccharide units. These materials are known as surfactants, but are not known as compatabilizers for ethoxylated nonlonic surfactants and detergent builders as they also function in the present invention.

Reducing saccharide containing 5 or 6 carbon atoms can be used, for example, glucose, factose and galactosyl moleties can substitute for the glucosyl moleties. Optionally, the hydrophobic group can be attached at the 2, 3, 4, etc. positions, thus giving a glucose or galactose as compared to a glucoside or galactoside. The intersaccharide bonds can be, for example, between the 1 position of the additional saccharide units and the 2-, 3-, 4- and/or 6 positions on the preceding saccharide units. Optionally, there can be a polyalkylene oxide chain joining the hydro-phobic molety and the polysaccharide molety. The

preferred alkoxide is ethylene oxide. Typical hydrophobic groups include alkyl groups, either saturated or unsaturated, branched or unbranched containing from about 8 to about 18, preferably from 10 to 16 carbon atoms. Preferably, the alkyl group is a straight chain saturated alkyl group. The alkyl group can contain up to 3 hydroxy groups and/or the polyalkoxide chain can contain up to about 10, preferably less than 5, most preferably 0, alkoxide moieties. Sultable alkyl polysaccharides are octyl, nonyl decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl and octadecyl, di-, tri-, tetra-, ponta- and haxaglucosides, glactosides, lactoses, factosides, glucoses, fructosides, fructoses and/or galactoses. Sultable mixtures include coconut alkyl, di-, tri-, tetra- and pentaglucosides and tallowalkyl tetra-, penta- and hexaglucosides.

The preferred alkyl polyglycosides have the formula

RO(C<sub>n</sub>H<sub>2n</sub>O)<sub>1</sub>(glycosyl)<sub>x</sub> wherein R is selected from the group consisting of alkyl, alkyl phenyl, hydroxy alkyl, hydroxy alkyl phenyl and mixtures thereof in which said alkyl groups contain from about 10 to 18, preferably from 12 to 14 carbon atoms; and is 2 or 3, preferably 2, t is from 0 to 10, preferably 0; and x is from 1-1/2 to 10, preferably from 1/2 to 3, most preferably from about 1.6 to about 2.7. The glycosyl is preferably derived from glucose. To prepare the compounds, the alcohol or alkylpolyethoxy alcohol may be formed first and then reacted with glucose, or a source of glucose to form the glycoside (attachment at the 1- position). The additional glycosyl units may be attached between their 1- position and the preceding glycosyl units 2-, 3-, 4 and/or 6 position, preferably predominantly the 2-position.

#### Aus. · · · Nonionic Surfactants

The ethoxylated nonionic surfactants which are suitable for use in the present invention include surface active or detergent compounds which contain an organic hydrophobic group and a hydrophylic group which is a reaction product of a solubilizing group such as carboxyl, hydroxyl, thiol, amide or amine with an alkylene oxide, such as ethylene oxide or propylene oxide or the polyhydration product thereof, such as polyethylene glycol and polypropylene glycol. Representative of such nonlonic surfactants are:

1. The polyethylene oxide condensates of alkyl phenols. These compounds include the condensation product of alkyl phenols having from 1 to 15, preferably 6 to 12 carbon atoms in a straight chain or branch chain configuration with from 4 to 25, preferably 4 to 18, moles of ethylene oxide per mole of alkyl phenol. The alkyl substituents in such compounds can be derived, for example, from polymerized polypropylene, dissobutylene and the like. Examples of compounds of this type include nonyl phenol condensed with about 9.5 moles of ethylene oxide per mole of phenol; dinonyl phenol condensed with about 15 moles of ethylene oxide per mole of phenol; dinonyl phenol condensed with about 15 moles of ethylene oxide per mole of phenol. Commercially available nonionic surfactants of this type include Igepal CO-630, marketed by the GAF Corporation, and Triton X-45, X-114, X-100 and X-102, all marketed by the Rohm & Hass Company.

2 The condensation products of aliphatic alcohols and thiols with from 1 to 25, preferably 5 to 16, moles of ethylene oxide. The alkyl chain with the aliphatic alcohol or thiol can either be straight or branched, primary or secondary and generally contain from about 8 to 22 carbon atoms. Examples of such ethoxylated alcohols include the condensation products of myristyl alcohol condensed with about 10 moles of ethylene oxide per mole of alcohol; and the condensation product of about 9 moles of ethylene oxide with coconut alcohol (a mixture of fatty alcohols with alkyl chains varying in length from 10 to 14 carbon atoms). A representative ethoxylated thiol is the condensation product of a C<sub>12</sub> tertiary thiol with about 7 moles of ethylene oxide per mole of thiol. Examples of commercially available nonionic surfactants of this type include Tergital 15-S-9, marketed by Union Carbide Corporation; Neodol - 45-9, Neodol 23-6.5, Neodol 45-7 and Neodol 25-7, marketed by Shell Chemical Company, Kyro EOB, marketed by The Proctor & Gamble Company and Alcodet ethoxylated thiols marketed by Alcolac Corporation. The ethoxylated alcohols and thiols are currently preferred nonionic surfactants.

3. The condensation products of ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The hydrophobic portion of these compounds has a molecular weight from about 1500 to 1800 and exhibits water insolubility. The addition of polyoxyethylene moieties to this hydrophobic portion tends to increase the water solubility of the molecule as a whole, and the squid character of the product is retained up to the point where the polyoxyethylene content is about 50% of the total weight of the condensation product, which corresponds to condensation of up to about 40 moles of ethylene oxide. Examples of compounds of this type include certain of the commercially

# PAGE 11/31 \* RCVD AT 12/15/2004 2:18:09 PM [Eastern Standard Time] \* SVR:USPTO-EFXRF-1/0 \* DNIS:8729306 \* CSID:704 331 7707 \* DURATION (mm-ss):10-20

available Pluronic surfactants, marketed by Wyandott Chemical Corporation.

4. The condensation products of ethylene oxide with a product resulting from the reaction of propylene oxide and ethylene diamine. The hydrophobic moiety of these products consists of the reaction product of ethylene diamine and excess propylene oxide, the moiety having a molecular weight from about 2500 to 3000. This hydrophobic moiety is condensed with ethylene oxide to the extent that the condensation product contains from about 40 to 80% by weight of polyoxyethylene and has a molecular weight from about 5000 to 11000. Examples of this type of nonionic surfactant include certain of the commercially available Tectronic compounds marketed by Wyandott Chemical Corporation.

70

#### Builders

The built detergent compositions of the invention also contain from 20 to 50%, preferably from 30 to 45%, and more preferably 35 to 45% by weight of a detergency builder, especially a water-soluble inorganic or organic electrolyte. Preferred electrolytes include the common alkaline polyvalent calcium ion sequestering agents. The builder can also include water-insoluble calcium ionic exchange materials.

Nonlimiting examples of suitable water-soluble inorganic detergent builders include alkaline metal carbonates, borates, phosphates, polyphosphates, bicarbonates, silicates, sulphates and chlorides. Specific examples of such salts include sodium and potassium tetraborates, perborates, bicarbonates, carbonates, tripolyphosphates, orthophosphates, pyrophosphates, hexametaphosphates and sulphates.

Examples of sultable organic alkaline detergency builders include water-soluble amino carboxylates and amino polyacetates, such as sodium and potassium glycinates, ethylene diamine tetraacetates, nitrilo triacetates and N-(2-hydroxy ethyl)nitrilo diacetates and diethylene triamine pentaacetates; water-soluble salts of phytic acid, such as sodium and potassium phytates; water-soluble polyphosphates including sodium, potassium and lithium salts of ethane-1-hydroxy-1, 1-diphosphonic acid, the sodium potassium and lithium salts of ethylene diphosphonic acid and the like; water-soluble polycarboxylates such as in salts of lactic acid, succinic acid, maioric acid, maleic acid, citric acid, carboxymethyloxy succinic acid, 1,1,2,2-ethane tetracarboxylic acid, cyclopentane-cls,cis,cis-tetracarboxylic acid, mellitic acid and pyromellitic acid; water-soluble organic amines and amine salts such as monoethanol amine, diethanol amine and triethanol amine and salts thereof.

Another type of detergency builder useful in the present composition comprises water-soluble material capable of forming a water-insoluble reaction product with water hardness cations preferably in combination with a crystallization seed which is capable of providing growth sites for said reaction product.

A further class of detergency builder materials useful in the present invention are insoluble sodium aluminosilicates, especially those having a calcium ion exchange capacity of at least 200 milligrams equivalent per gram and a calcium ion exchange rate of at least .53 grams per litre per minute per gram.

## Additional Ingredients

The built detergent compositions of the present invention can be supplemented by the usual additives conventionally employed in detergent compositions. Optional Ingredients Include soil suspending agents at about 0.1 to 10% by weight, including water-soluble salts of carboxy methyl cellulose, carboxy hydroxy methyl cellulose and polyethylene glycole having a molecular weight of about 400 to 10000. Dyes, pigments, optical brighteners and perfumes, enzymes, preservatives such as sodium benzoate, alkaline metal or alkaline earth metal siticates, suds regulating or suppressing agents, natural and synthetic microcrystalline and oxidized microcrystalline waxes, inorganic and organic peroxy bleaching agents, polyphosphonic acids and acid salts. These materials may be employed in the practice of this invention at conventional levels normally employed in detergent formulations.

Substantially any of the known anionic fluorescent brightening and/or weiting agents can be employed in the practice of this invention. The currently most preferred whiteners are 4,4'-bls[(4-anilino-6-[bls(2-hydroxy ethyl)-amino]-s-triazine-2-yl)amino]-2,2' stilbene disulphonic acid; 4,4-bis[(4-anilino-t[N-2-hydroxy ethyl-N-methyl amino]- s-triazine-2-yl)amino]-2,2' stilbene disulphonic acid disodium salt; and 4-[2+naphthol(1,2-d)triazol-2-yl]-2-stilbene sulphonic acid sodium salt. Also suitable for use are nonionic brighteners such as p-[3-(p-fluorophenol)-2-pyrozolin-1-yl] benzene sulfonamide.

The compositions of the present invention can also optionally include both cationic and anionic

surfactants. Substantially any of the known cationic surfactants can be advantageously included in the detergent compositions of the invention. Preferred cationic surfactants are the alkyl quaternary ammonium halides and sulphates, especially the mono long chain alkyl ( $C_{8-22}$ ) and alkylene oxylated mono long chain alkyl surfactants. The most preferred quaternary ammonium surfactants are the chloride, bromide and methyl sulphate  $C_{8-22}$  alkyltrimethylammonium salts,  $C_{8-22}$  alkyl (dihydroxyethyl) methylammonium salts,  $C_{8-22}$  alkyloxypropyl (dihydroxyethyl) methylammonium salts,  $C_{8-22}$  alkyloxypropyl (dihydroxyethyl) methylammonium salts and  $C_{8-22}$  alkyloxypropyl (dihydroxyethyl) methylammonium salts and  $C_{8-22}$  alkyloxypropyl (dihydroxyethyl) methylammonium salts being especially preferred. The current cationic surfactant of choice is isotridecyloxypropylbis (hydroxyethyl) methyl ammonium chloride. The cationic surfactants, when used, will be present in amounts up to 12 percent by weight, preferably from 1 to 5 percent by weight, and most preferably in the range from 2 to 4 percent by weight.

Although substantially any of the known anionic surfactants which are typically employed in detergent compositions can be advantageously included in the detergent compositions of the invention, water-soluble salts, preferably the alkaline metal ammonium and alkylol ammonium salts, of organic sulphuric reaction products having in their molecular structure an alkyl group containing from about 10 to 20 carbon atoms and a sulphoration or sulphuric acid group are especially preferred. Examples of this group of anionic surfactants are the sodium and potassium alkyl sulphates, especially those obtained by sulphating the higher alcohols (C8-C18 carbon atoms) such as those produced by reducing the glycerides of tailow or coconut oil; and the sodium and potassium alkyl benzene sulphonates in which the alkyl group contains from about 9 to about 15 carbon atoms, in straight chain or bent chain configuration. Especially valuable are linear straight chain alkyl benzene sulphonates in which the average number of carbon atoms in the alkyl group is from about 11 to 13, abbreviated as C11-C13 LAS.

Other suitable anionic surfactants are the alkyl polyethoxylate sulphates, particularly those in which the alkyl group contains from 10 to about 22, preferably from 12 to 18 carbon atoms, and wherein the polyethoxylate chain contains from 1 to 15 ethoxylate moieties, preferably from 1 to 3 ethoxylate moieties. Also suitable for use in the invention are anionic surfactants such as the sodium alkyl glycerol ether sulphonates, especially those ethers of higher alcohols derived from tallow and coconut oil, sodium coconut oil, fatty acid monoglyceride sulphonates and sulphates; sodium and potassium salts of alkyl phenol, ethylene oxide ether sulphates containing from 1 to 10 units of ethylene oxide per molecule and wherein the alkyl groups containing from 1 to 10 units of ethylene oxide per molecule and wherein the alkyl group contains from 10 to 20 carbon atoms.

Other anionic surfactants include water-soluble salts of esters with alpha-sulphonated fatty acids containing from 6 to 20 carbon atoms in the fatty acid group and from 1 to 10 carbon atoms in the ester group; water-soluble salts of 2-acytoxy-alkane-1-sulphonic acids containing from 2 to 9 carbon atoms in the acyt group and from 9 to 23 carbon atoms in the alkane molety, alkyl ether sulphates containing from 10 to 20 carbon atoms in the alkyl group and from 1 to 30 moles of ethylene oxide: water-soluble salts of olefin sulphonates containing from 12 to 24 carbon atoms, and beta-alkyloxy alkane sulphonates containing from 1 to 3 carbon atoms in the alkyl group and from 8 to 20 carbon atoms in the alkane moiety. When used, the anionic surfactants will be employed in amounts up to 12 percent by weight, preferably in the range from 1 to 7 percent by weight, and most preferably, from 2 to 5 percent by weight. The anionic surfactants are preferably employed in combination with cationic surfactants. Alkyl sarcosinates are currently preferred anionic surfactants.

Other optional ingredients which can be included in the detergent compositions of the present invention, in their conventional art established levels for use include solvents, bleaching agents, bleach activators, soils suspending agents, corrosion inhibitors, dyes, fillers, optical brighteners, germicides, pH adjusting agents (monoethanolamine, sodium carbonate, sodium hydroxide and the like), enzymes, enzyme-stabilizing agents, performes, fabric softening components, static control agents, and the like.

The liquid detergent compositions of the present invention can be conveniently prepared in accordance with the known formulation procedures or techniques which are usually employed in the preparation of conventional anionic surfactant-based liquid detergent products. Preferably, the desired surfactant components (i.e., the glycoside surfactant, the ethoxylated nonionic surfactant, and, if employed, the anionic and/or cationic surfactant components) are first dissolved in water and any auxiliary additives which may be desired for use herein such as, for example, stabilizers, perfumes, preservatives, colourants, etc. are subsequently added to the resulting aqueous surfactant solution. Preferably, any such auxiliary ingredients either will be water soluble in character or will be presolubilized or pre-dispersed in a portion of the surfactant system prior to being added to the mixture. The application of heat to the aqueous mixture is often beneficial during the formulation process in facilitating the obtaining of a homogeneous mixture and it

is generally preferred to stir, or to otherwise provide good agitation to the mixture during said process. Further, in those formulations employing a viscoeifying agent such as ammonium or alkali metal ealts, it is generally preferred to dissolve the desired amount of same in the water at the beginning of the formulation process.

The pH of the present formulations is not particularly critical and can be varied or adjusted as desired in a given instance. As a general rule, however, the pH of such formulations will typically be within the range of from about 8 to 12.

#### Experimental Procedures and Results

The detergent compositions were evaluated in the examples as follows:

Soiled cloths were washed in a laboratory-scale washing machine (Terg-O-Tometer, U.S. Testing Company) which simulates the action of an agitator-type home washer. Appropriate amounts of detergent formulations are added to one litre of water at a controlled hardness. Wash temperature, agitator speed and wash, as well as rinse, time can be controlled via the Terg-O-Tometer. After washing, rinsing (machine or by hand) and drying, detergency is taken as a change in the reflectance of the cloths. Assignments of formulas to the test spots are randomized using a simple random number table.

The effectiveness of the detergent compositions is determined by reflectance readings using a Gardner Color Difference Meter, with all cloths being read before and after laundering. Each cloth is individually placed on the reflectometer and covered by a white ceramic plate standard. For the Bandy black clay soll cloths, only Rd readings are taken. For all cloths, a and b values, in addition to Rd readings, may be observed.

The soil types which were evaluated are as follows:.

Bandy Black Clay (BBC): an artificially prepared soil cloth prepared by a dry soiling method where the clay is ball milled into the fabric.

TFI: a printed soil cloth (mineral oil-carbon black base) purchased from Test Fabrics Incorporated.

HCO: a soiled cloth prepared by immersion in a lard, margarine and peanut oil-carbon black mixture.

 Spangler (SPNG): a soiled cloth prepared by immersion in a soil bath (synthetic sebum, air conditioner dust) and then padded and dried.

EMPA: a soiled cloth prepared by immersion in an olive oil-carbon black mixture.

DMO: a soiled cloth prepared by immersion in dirty motor oil.

Cotton, cotton/Dacron, cotton with permanent press finish, cotton, Dacron with permanent press finish were used for the Bandy Black Clay, TFI, DMO, HCO and Spangler soils. Cotton and cotton: Dacron with permanent press finish were used for EMPA and grass soils.

The following examples illustrate, but are not limiting of, detergent compositions of the present invention. All formulations were evaluated at \$7.7°C. (100°F.) in water having 3, 6 or 12 grains of hardness.

Stability evaluations were made by storing containers of the detergent compositions and evaluating the appearance of the compositions over time. Compositions which had phase separated, that is, formed distinct layers, over the indicated test periods, were considered to be failures and thus unacceptable. The failures are reported as "F". The formulations which over the test period had not phase separated but exhibited some loss in homogeneity of the emulsion, e.g., creaming, were reported by the symbol "S". These formulations while not optimal, are still satisfactory for use. Formulations which exhibited little, if any,

change over the test period were reported by the symbol "P".

EXAMPLE 1			
	Ілуе	Control	
Tetrapotassium pyrophosphate  C12-12 Alkylmonoglyceride  C12 t-thiol ethoxylate, 7 moles ethylene oxide Isotridecyl oxypropyl-bis(2-hydroxy ethyl)-methyl ammonium chloride Sodium lauryl sarcosinate Sodium lauryl sulfate Water	40.0 7.0 7.0 2.0 0.0 1.8 Q.S.	40.0 7.0 7.0 2.0 2.4 0.0 Q.S.	77.
Test Results - Reflectance Difference (before and after washing readings)			
TFI-31 SPNG-31 BBC-31 HCO-31	25.20 16.50 86.60 7.40 13.60	24.80 15.40 87.80 7.00 10.40	22.0 24.8 87.90 7.20 7.20
DMO-31	10.00	19.90	1 /.20

1: Cloths were washed in water having a hardness of .051 grams/I (3 grains/U.S. gallon).

2: Cloths were washed in water having a hardness of .205 grams/I (12 grains/U.S. gallon).

As can be seen from the reflectance data, detergent compositions embodying the invention, on balance, provide equivalent to better detergency results on oil-based soils when compared to a leading commercial liquid detergent, in both "soft" and "hard" water, with performance of both the invention detergents and the commercial detergents being less effective in the harder water. The concentrated detergents of the invention were used at 1/4-cup dosage equivalent and the commercial detergent was used at 1/2-cup dosage equivalent, i.e., 0.11% and 0.18%, respectively, as the actual concentrations in the wash water.

#### **EXAMPLE 2**

Formulations embodying the inventive concept were made up as set torth in the Table identified as Example 2. As can be seen from the reflectance data of Examples 1 and 2, detergent compositions embodying the concept of the invention can be expected to provide equivalent to better performance to that provided by the commercial liquid detergent control of Example 1 on most oil-based stains, and to provide an acceptable level of performance on sebum (SPNG) stains.

## **EXAMPLE 3**

Formulations embodying the inventive concept were made up as set forth in the Table identified as Example 3. The reflectance data reported in the Example 3 Table demonstrate that the detergent compositions of the invention can be formulated, e.g., increase alkalinity, to increase the level of performance on sebum stains and it is not expected that the increase in sebum stain removal would be accompanied by any loss in oil-based stain removal.

## **EXAMPLE 4**

Formulations embodying the inventive concept were made up as set forth in the Table identified as Example 4. The data is generally self-explanatory. The data reported in the Example 4 Table demonstrate that excellent to acceptable emulsions are provided by the invention formulations. The data also show that emulsion stability may be a factor of the alkylglycoside:nonionic ratio, which preferably is 1:1.

## EXAMPLE 5

Formulations embodying the inventive concept of the invention were made up as set forth in the Table identified as Example 5. The data is generally self-explanatory. The stability data reported in the Example 5 Table demonstrate that excellent to acceptable emulsions are provided by the invention formulations. The stability results reported as an "S" are indicative of emulsions which remained acceptable over the test period but showed slight separation or creaming over the period.

## EXAMPLE 6

Formulations embodying the inventive concept were made up as set forth in the Table identified as Example 6. The stability data reported in the Example 6 Table demonstrate that the inclusion of additives, such as antiredeposition agents, can offset the stability of the Invention detergents and that some additives may have a detrimental effect. Polyethylene glycol is a preferred antiredeposition agent in the formulations of this invention. Thus, some experimental work of a non-inventive nature may be required in developing fully formulated detergent compositions.

# EXAMPLE 7

Formulations embodying the inventive concept were made up as set forth in the Table identified as Example 7. The stability data reported in the Example 7 Table demonstrate the effect of changing the nonionic sufactants in the invention formulations. The data Indicates that a broad range of ethoxylated nonionic sufactants may be used to prepare detergent compositions in accordance with the invention, and that some experimental work of a non-inventive nature may be required in optimizing the stability of fully formulated compositions.

EP 0 405 967 A2

Ī	Ð	CAMPLI	E 2						
ľ		91	93	98	96	94	97	95	92
ł	Potassium tripolyphosphate	38.5	36.5	36.5	36.5	36.5	36.5	36.5	36.5
5	C12-12 Alkyl triglycoside	7.0	7.0						
	C <sub>12-13</sub> Alkyl monoglycocide		l l	7.0	7.0	7.0	7.0	7.0	7.0
	C12 t-thiol ethoxylate, 7 moles ethylene oxide	7.0	7.0						
	nonionic C <sub>12-15</sub> -O-{CH <sub>2</sub> CH <sub>2</sub> O) <sub>7</sub> H nonionic	. •			7.0				
10	C12-13-O-(CH2CH2O)sH nonionic		!			7.0			
	C11-O-(CH2CH2O)sH topped nonlonic		i	l i				7.0	
	C <sub>12</sub> sec-thiol ethoxylate, 9 moles ethylene		7.0	[					7.0
	oxide nonionic		l				}		
	C <sub>12-14</sub> -O-(CH <sub>2</sub> CH <sub>2</sub> O) <sub>5</sub> H narrow, nonionic		1			<b>'</b>	7.0	ļ	
15	Isotridecyloxypropyl-bis(2-hydroxyethyl)methyl	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
	ammonium, chloride	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4
	Sodium lauryl sarcosinate	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.
	Water	u.s.	- G.S.	، -دینه	4.0,	0	4.0	1	1.724.7
20	Test Results: Reflectance Difference				<b>.</b>	<u> </u>		·	
	TFI-61	20.5	19.6	18.0	18.4	19.1	19.8	20.2	20.0
	SPNG-61	12.7	13.2	13.B	14.4	12.8	11.8	14.8	13.9
	EMPA-61	10.1	13.0	12.2	12.2	10.6	9.7	10.4	13:1

<sup>1:</sup> Cloths were washed in water having a hardness of 102 grams/l (6 grains/gallon).

E)	KAMPLI	E 3	•	· ,	•			
	01	02	03	04	05	06	07	08
Potassium tripolyphosphate	36.5	36.5	36.5	36.5	.36.5	36.5	36.5	36.5
Sodium carbonate	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5
Alkyl monoglycoside	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
C <sub>11</sub> -O-(CH <sub>2</sub> CH <sub>2</sub> O) <sub>7</sub> H nonionic	7.0	1	1	•				
AOS anionic		7.0				Į.		l
AES anionic <sup>2</sup>			7.0 :	2 .		İ	*: **.	٠.
G <sub>11</sub> -O-(CH <sub>2</sub> CH <sub>2</sub> O)₅ H topped nonionic ·		1	1	13.0		1	1	
C <sub>11</sub> -O-(CH <sub>2</sub> CH <sub>2</sub> O) <sub>3</sub> H nonionic	i				13.0	ļ		
C <sub>12</sub> t-thiol ethoxylate, 7 moles ethylene oxide		İ	ľ	ļ		13.0		١.
nonionic	İ	1	i		1	ļ		1
C <sub>12</sub> sec-thiol ethoxylate, 9 moles ethylene	ł		i		i	l	18.0	
oxde nonionic		1			١.	1		١.
	' '						i	13.0
	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Isotridecyloxypropyl-bis(2-hydroxyethyl)methyl	2.0	2.0	2.0	- 2.0				
ammonium chloride	1 24	ł		1			1	
Sodium lauryl sarcosinate	2.4	Q.S.	a.s.	a.s.	a.s.	a.s.	Q.S.	Q.S
Water	a.s.	u.5.	u.s.	w.s.	u.s.	4.5.	2.3.	- W-
Test Results: Reflectance Difference								
SPNG-63	19.0	16.3	16.4	20.1	21.9	18.9	21.4	19.

<sup>1:</sup> a - olefin sulfonate anionic

\_\_ 2: alkyl ether sulfonate anionic

<sup>3:</sup> Cloths were washed in water having a hardness of .051 grams/I (6 grains/gallon).

	EXAMPLE 4															
		51	52	53	54	55	56	57	58	69	61	62	63	64	65	66
	TKPP <sup>3</sup>	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0
5	APG-1 <sup>b</sup>	5.0	7.5	3.75	5.0	5.0	5.0	5.0	5.0	5.0	7.5	7.0	7.0	7.0	7.0	7.0
	TTE-7°	7.5	2.5	7.5	5.0	10.0	10.0	5.0		5.0	2.5	7.0	7.0	7.0	7.0	7.0
	TTE-9d								5.0						<u> </u>	
	Quat <sup>e</sup>	20	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0	3.0	4.0	2.0	2.0
	Sarci	2.4	24	24	1.8	1.5	2.4	2.4	2.4	0.8	0.0	0.0	2.4	2.4	2.4	0.0
10	LES"										1.2	1.2				1,2
ļ	H <sub>2</sub> O	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.Ş.	Q.S.
	Stability Test							1	ļ							1
	Results															
75	1 mo. stability	F	F	F	P	F	F	Р	P	P	F	P	P	P	Þ	P
	3 mo. stability	F	F	F	Р	F	F	P	P	F	F	P	P	P	P	7

a: Tetrapotassium pyrophosphate

b: C<sub>12-13</sub> Alkyl polyglycoside having 1 mole of glycoside

c: C12 Tertiary thiol ethoxylate having 7 moles of ethylene oxide

d: C12 Tertiary thiol ethoxylate having 9 moles of ethylene oxide

e: isotridecyloxypropylbis(2-hydroxyethyl)methyl ammonium chloride nonionic

f: Lauryl sarcosinate sodium salt anionic

g: Lauryl ether sulfate sodium salt anionic

			Ð	CAMPLE	<u>5</u>				
•	67	68	71	72	73	74	75	76	78
TKPP	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0
APG-1	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
TTE-7	7.0	7.0			7.0	7.0	7.0	7.0	7.
25-7°				7.0					
1-512			7.0	•		İ			
Quat	2.0	2.0 .	2.0	2.0	2.0			2.0	2.5
Sarc		2.4	2.4	2.4	2.4	2.4	0.0	3.0	2.
LES	1.8								•
Water	Q.S.	Q.S.	Q.S. ·	. C2.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S
Stability Test Results									
1 mo. stability	P	P	P	Р	P	?	Р	P	P
3 mo. stability	?	3	?	?	?	?	?	?	?

1: C12-15 ethoxylated alcohol having 7 moles of ethylene oxide

2: Topped ethoxylated C11 alcohol having 5 moles ethylene oxide

. —			EXAM	PLE 6				
	B1	82	83	84	85	86	B7 ·	88
KIP .	\$0.0	.30.0	30.0	30.0	30.0	30.0	36.5	
K Citrate								38.6
APG-1 -	7.0	7.0	7.0	7.0	. 7.0	7.0	⋯7.0	7.0
TTE-7	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
Quat	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Sarc	24	2.4	2.4	2.4	2.4	2.4	2.4	2.4
PA-1000 <sup>2</sup>		1.0		ţ		Ì	]	1
PA45003	1.0			İ	l	l	1	
SSMA-10004		i	1.0	[			1.0	1.0
PEG-5000 <sup>5</sup>	j		1	1.0		1	1.0	1.0
<b>b</b> Abe		1		1	1.0		1	ł
CHC/MC7 .		1	1	) ·		1.0		
H <sub>2</sub> Q	Q.S.	Q.S.	- a.s.	Q.S.	Q.S.	Q.Ş.	Q.S.	Q.S.
Stability Test	1	ļ	1	i i	\ ·	1	l l	
Results	:	1			٠.	ŀ		i
	-	?	P	Р	F	F	P	P
1 mg. stability	F.	F	F	P	<b>#</b>	F	F	F
3 mo. stability	r		1 -		<del>  '</del>	<u> </u>	ــــــــــــــــــــــــــــــــــــــ	ــــــــــــــــــــــــــــــــــــــ

- 2: Polyacrylate homopolymers, 1000 mw
- 3: Polyacrylate homopolymers, 4500 mw
- 4: Sulphonated styrene maleic anhydride polymer, 1000 mw
- 5: Polyethylene glycol, 5000 mw
- 6: Polyvinyl pyrollidone
- 7: Mixture of carboxymethyl cellulose and methyl cellulose

٠								
	•		EXAM	PLE 7	•			
	91	92	93	94	95	96	97	98
КТР	36.5	36.5	36.5	36.5	36.5	36.5	36.5	36.5
APG-3	7.0		7.0					
APG-1		7.0		.7.0	7.0	7.0	7.0	7.0
TTE-7	7.0	i						···7.0
STE-91		7,0:	7.0 -	1				٠
23-5 <sup>2</sup>		ĺ		7.0	l	·		ļ
1-51	,				7.0	·	}	
25-7	Ì	1	ļ · ·	<b>!</b> "		7.0	i '	
24-25N <sup>3</sup>			İ		,		7.0	
Quat	2.0	2.0	2.0	2.0	2.0	. 2.0	2.0	2.0
Sarc	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4
H <sub>2</sub> O	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.S.	Q.Ş.
Stability Test Results								
1 mo. stability	P ?	P	P ?	? F	? F	P P?	P	P ?

- 1: C12 Secondary thiol ethoxylate having 9 moles of ethylene oxide
- 2: C12-13 alcohol having 5 moles ethylene oxide
- 3: Narrow C<sub>12-14</sub> alcohol having 5 moles ethylene oxide

15

20

30

			EX	AMPLE	8	•			
	01	02	03	04	05	06	07	08	09
KTP	36.5	36.5	36.5	36.5	38.5	36.5	36.5	36.5	36.5
APG-1	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
1-71	7.0								
AOS		7.0				•			
AES `			7.3		i				
1-51 .			-	13.0	'		İ	1	١.
1-9					13.0		1	į į	•
TTE-7						13.0	٠	i	
STE-0		]	1	1			13.0	}	<b> </b>
24-60N3			ļ					13.0	
24-85N4				ļ	}	ŀ			13.0
Quat ·	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Sarc	2.4		ĺ						
H₂Ó	Q.S.	Q.S.	Q.S.	Q.S.	Q.Ş.	Q.S.	Q.S.	Q.S.	Q.S
Stability Test	1					Ĺ			
Results						1			
1 mo. stability	Р	P	Þ	P	P	P	P	F	F
3 mo. stability	7	P	F	F	F	?	?	F	F

- 1: C11 alcohol having 7 moles ethylene oxide
- 2: C11 alcohol having 9 moles ethylene oxide
- 3: Narrow c12-14 alcohol having 7 moles ethylene oxide
- 4: Narrow C12-14 alcohol having 9 moles ethylene oxide

The data demonstrate the effect of changing the nonionic surfactants in the invention formulations, and indicates that experimental work of a non-inventive nature may be required in developing fully formulated compositions.

## Claims

10

- 1. A liquid detergent composition comprising from 5 to 22 percent by weight of at least one ethoxylated nonionic surfactant, from 3 to 11 percent by weight of at least one alkyl polyglycoside and from 20 to 50 percent by weight of at least one detergent builder, and soft water.
  - 2. A liquid detergent composition according to claim 1 wherein said ethoxylated nonionic surfactant is present in a range from 5 to 12 percent preferably 7 percent, by weight, said alkyl polyglycoside is present in an amount in the range from 4 to 9 percent, preferably 7 percent, by weight and said builder is present in an amount in the range from 30 to 50 percent, preferably 35 to 45 percent, by weight.
- 3. A liquid detergent composition according to claim 1 or 2 containing up to 12 percent, preferably 1 to 5 percent and more preferably 2 to 4 percent, by weight of at least one cationic surfactant.
- A liquid detergent composition according to claim 3 wherein the cationic surfactant comprises isotridecyloxypropylbis (2-hydroxyethyl) methyl ammonium chloride.
- 5. A liquid detergent composition according to any of claims 1 to 4 containing up to 12 percent, preferably in the range from 1 to 7 percent, more preferably in the range of 2 to 5 percent, by weight of at least one anionic surfactant.
- 6. A liquid detergent composition according to claim 5 wherein the anionic surfactant comprises sodium lauryl sarcostinate.
- 7. A liquid detergent composition according to any of claims 1 to 6 containing from 0.5 to 3 percent by weight of polyethylene glycol having a molecular weight in the range from 2000 to 20000.



Europäisches Patentamt European Patent Office Office européen des brevets

(1) Publication number:

0 405 967 A3

**②** 

# **EUROPEAN PATENT APPLICATION**

- (1) Application number: 90307071.2
- 2 Date of filling: 28.06.90

(i) Int. Cl.5; C11D 1/825, C11D 1/835, C11D 1/83, C11D 1/86

- @ Priority: 30.06.89 US 373912
- ② Date of publication of application: 02.01.91 Bulletin 91/01
- Designated Contracting States:

  AT BE CH DE ES FR GB IT LI LU NL SE
- Date of deferred publication of the search report:
  17.04.91 Bulletin 91/16
- Applicant: Amway Corporation 7575 East Fulton Road Ada Michigan 49355(US)
- inventor: Flower, David Michael
   7232 Mount Ash, S.E.
   Grand Rapids Michigan 49506(US)
- Representative: Robinson, Anthony John Metoalf et al Kilburn & Strode 30 John Street London, WC1N 2DD(GB)
- Built liquid detergent compositions.
- ② Liquid laundry detergents comprising at least one ethoxylated nonionic surfactant, at least one alkyl polyglycoside and at least one detergent builder display excellent shelf stability, notwithstanding the fact that such compositions contain from 20 to 50 percent by weight of builder and from 5 to 22 weight percent by ethoxylated nonionic surfactant.

EP 0 405 967 A

Xerox Capy Centre



# EUROPEAN SEARCH . REPORT

Application Number

EP 90 30 7071

D	OCUMENTS CONSI				
Category		n indication, where appropriate, vent passages		Rejevant to etaim	CLASCIFICATION OF THE APPLICATION (Int. CL5)
х	EP-A-0 075 995 (THE PRO * claims: page 13, lines 18-2 lines 1-26; page 20, line 34	3; page 15, lines 9-35; pa		1-3,5	C 11 D 1/66 C 11 D 1/825 C 11 D 1/835
x	EP-A-0 106 692 (THE PRO " whole document "	OCTER & GAMBLE CO.)		1-3,5,7	C 11 D 1/88 C 11 D 1/83
<b>x</b>	WQ-A-8 604 349 (STALEY "whole document"	MANUFACTURING CO.)		1,2,7	
Α '	EP-A-0 105 556 (THE PRO * claims *	OCTER & GAMBLE CO.)		1,2,5	
A	WO-A-8 602 943 (STALEY claims; example 2	MANUFACTURING CO.)		1,2,5,6	
A	WO-A-8 706 949 (STALEY	MANUFACTURING CO.)		1,2	
A	WO-A-8 809 369 (STALEY " claims; page 10, lines 2-6; lines 1-15 "			1,2,7	TECHNICAL FIELDS SEARCHED (Int. Cl.6)
A	US-A-4 488 981 (A.D. URF "summary; column 4, lines claims "			1,2,5	C 11 D
	,			•	•
	The present search report has t	pen drawn up for all cisims			•
	Place of search	Date of completion of s	earch		Examiner .
	Berlin	10 January 91			PELLI-WABLAT B
Y: A: O:	DATEGORY OF ORED DOCU particularly relevant if taken alone particularly relevant if combined wit document of the same catagory technological background non-written disclosure intermediate document		the fili D: docum L: docum	ng date ent cited in th out cited for d er of the same	